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MANUFACTURING TECHNOLOGY FOR SPICE OLEORESIN MICROCAPSULES
[Xiang xin liao you shu zhi wei jiao nang de sheng chan gong yi]

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Claims

1. Manufacturing technology for spice oleoresin microcapsules, characterized in that the manufacturing technology comprises:

A. Raw material preprocessing: After selection and cleaning of superior quality raw materials, they are crushed into a fine powder or paste, and filtered to await use;

B. Oleoresin extraction: The above-described raw material powder or paste is extracted at room temperature using ethanol as a solvent to perform continuous percolation and extraction, or CO_2 supercritical fluid extraction is used, to obtain the anhydrous or hydrous oleoresin;

C. Emulsification and encapsulation: At proportions of anhydrous oleoresin:edible gum:water = 1:1-3:5-10, the first emulsification is crude emulsification, which is then input into a homogenizer to undergo homogenization and emulsification 1-2 times at 10-40 MPa, to serve as the O/W emulsion;

D. A spray gun is used to spray the homogenized and emulsified emulsion into a low-temperature atomization dryer for dehydration and drying, the dryer's inlet air temperature is 80-100°C and the outlet air temperature is 40-60°C, thus a powdered oleoresin microcapsule product with a grain diameter 10-70 μm is obtained; or further undergoes:

E. Pelleting: The powdered microcapsules obtained using D serving as the medium, are then placed into an atomized dryer fluidized bed granulation chamber, the homogenized emulsion is suspended, adsorbed and spray-atomized, after drying the output obtained is a granular microcapsule product with a grain diameter 80-110 μm.

2. Manufacturing technology for spice oleoresin microcapsules according to Claim 1, characterized in that the described CO_2 supercritical fluid extraction used sends the powder or paste to the extraction chamber for extraction of the mixture with supercritical fluid CO_2 at 32-36°C and 25-35 MPa for 60-120

min, high-pressure separation of the liquid and residue is performed; the residue is discharged into a residue storage tank, and fluid CO_2 containing the extracted oleoresin is then placed into a vacuum separation chamber at 0.1-0.3 MPa to separate the liquid and the gas; gaseous CO_2 is recovered and, after processing, sent for use in the next cycle, and the oleoresin is precipitated out and sent to the storage device until use.

3. Manufacturing technology for spice oleoresin microcapsules according to Claim 1, characterized in that the described spray gun used sprays the homogenized and emulsified emulsion into a low-temperature atomization dryer for dehydration and drying; the spray gun's spray input pressure is 0.2-0.3 MPa and the spray nozzle aperture is 0.8-1.2 mm.

4. Manufacturing technology for a spice oleoresin microcapsule according to Claim 1, characterized in that the described edible capsule that serves as a wall material is two or more of acacia gum, maltodextrin, xanthan gum and soluble starch mixed according to a fixed proportion to form an edible gum.

The present invention pertains to a spice flavoring product manufacturing technology in the food /1*
product industry, in particular it is a technology for manufacturing spice oleoresin microcapsules, especially suited for purifying garlic, fresh ginger, paprika, Sichuan peppers and other natural spice flavoring products for more convenient use. It also facilitates preservation of the flavoring in powder or grain form.

At present, there has been greater and greater attention paid to natural spice flavoring products frequently used for cooking food and in the food industry that have undergone microencapsulation processing for direct use in cooking and food manufacturing to thus achieve improved resource utilization rates, extend a condiment's preservation (freshness) period, make it convenient to use, and other objectives. Microencapsulation processing of the above-described flavoring products mainly uses two methods. The first is extraction and purification of natural spice volatile oils, which are the main taste agents, to serve as the core material, then said purified oil and the wall material are mixed, homogenized, emulsified, encapsulated and finally undergo high-temperature atomization drying to prepare the corresponding purified oil microcapsules. Articles published by Lin Bo et al. "Study of High-Pressure Purifying Process to Prepare Microcapsules" in Chemistry World (1990 Volume 8, pp. 351-353), Xiang Yunfeng et al. "Experimental Research on Garlic Oil Microcapsule Preparation Technology" in Food Industry (1995 Volume 5, pp. 29-31), and Chen Shusheng et al. "Microcapsule Technology Applications in Flavoring Products" in Food Research and Development (1995 Volume 3, pp. 22-25) are all related to this type of manufacturing method. In this technology, the inadequacies of using purified oil as the core material and high-temperature atomization drying to prepare the microcapsules lie in that the loss of purified oil and its volatile substances is great and the retention rate is low; high-temperature drying damages some compounds and generates new compounds that adversely affect the taste and oral sensation and result in poor product quality; in addition, there is also an odor

* [Numbers in right margin indicate pagination of the original text.]

present in the raw material during the purified oil extraction process. Further, it is hard to fully extract the taste ingredients, the yield is low, the raw material is not fully used, high-temperature drying requires the appropriate high-pressure cooker, equipment investment is high, and there are other defects. The second method uses oleoresin as the core material. Because oleoresin contains the purified oil of the represented spice flavor and it also contains higher boiling point sesquiterpenoids and resins and natural antioxidant ingredients of the spice flavor represented, it has good antioxidant ingredient release capabilities, its taste is soft and mellow, its fragrance and spice flavor are in harmony, the storage period is long and the effective ingredients in the spices can be completely extracted, as well as other advantages. R. Zilberboim et al. "Microencapsulation by a Dehydrating Liquid: Retention of Paprika Oleoresin and Aromatic Esters" published in the Journal of Food Science (1986 Vol. 51, No. 5, pp. 1301-1306) relates to this technology. Although said technology possesses numerous advantages for using oleoresin as the core material in the manufacture of microcapsules, it only uses acacia gum as the wall material, after undergoing homogenizing, emulsifying and encapsulation, it first undergoes anhydrous ethanol dehydration, then it undergoes vacuum drying to produce the microcapsules. Here, /2 when the final concentration of acacia gum reaches 40% and the anhydrous ethanol and emulsion liquid proportion is 10:1, the core material retention rate only reaches 83%, additionally it is necessary to add ethanol recovery and purifying equipment; and when vacuum drying the microcapsule is extremely susceptible to quick adhesion and hard to form into a powder or grain, which affects product quality. Thus, said process additionally has high production costs, large accessory equipment investments, poor powder and grain size, product quality that is hard to guarantee, and other drawbacks.

The objective of the present invention is to use oleoresin as the core material in manufacturing microcapsules and to preserve its numerous advantages, thus fundamentally improving retention rates and product quality, reducing manufacturing costs, improving resource use rates, and saving energy.

The solution in the present invention is to continue using routine industrial processes in the prior art for preprocessing raw materials, then the preprocessed raw material is extracted using supercritical fluid CO_2 or 35-95% ethanol as a solvent at room temperature to perform continuous percolation to prepare the oleoresin; and a fixed proportion of membrane-forming edible gums, such as acacia gum, maltodextrin, xanthan gum, soluble starch and others that are readily soluble in water, are used as the emulsified wall material for the crude emulsion, homogenized at room temperature and 10-40 MPa for the O/W emulsion and encapsulation, finally undergoing low-temperature atomization drying to prepare powder or grain microcapsules. Thus, the manufacturing technology for the present invention comprises:

A. Raw material preprocessing: After selection and cleaning of superior quality raw materials, they are crushed into a fine powder or paste, and filtered to await use;

B. Oleoresin extraction: For the above-described raw material powder or paste, ethanol at room temperature is used as the solvent to perform continuous percolation and extraction, or supercritical fluid CO_2 extraction is used, to prepare the anhydrous or hydrous oleoresin;

C. Emulsification and encapsulation: At proportions of anhydrous oleoresin:edible gum:water = 1:1-3:5–10, the first emulsification is the crude emulsification, then it is sent to a homogenizer at 10-40 MPa, undergoes homogenization and emulsification 1-2 times to be the O/W emulsion;

D. Dehydration and drying: A spray gun is used to spray the homogenized and emulsified emulsion into a low-temperature atomization dryer for dehydration and drying, the dryer's air inlet temperature is 80-100°C and the air outlet temperature is 40-60°C, thus preparing a powdered oleoresin microcapsule product with a grain diameter 10-70 μm;

E. Pelleting: The powdered microcapsules obtained in D serving as the medium, are placed into an atomization dryer fluidized bed granulation chamber, suspended, adsorbed and spray-atomized into the

homogenized emulsion, after drying the output is a granular microcapsule product with a grain diameter of 80-110 μm.

The supercritical fluid CO_2 extraction above is used to prepare anhydrous or hydrous oleoresin, which means the substance powder or paste is sent to the extraction chamber at 32-36°C and 25-35 MPa /3 For extraction of the mixture with supercritical fluid CO_2 mixture for 60-120 min, high-pressure separation of the liquid and the residue; the residue is discharged to the residue storage tank, and the fluid CO_2 containing the extracted oleoresin is then placed in a vacuum chamber at 0.1-0.3 MPa pressure to separate the liquid and the gas. Here gaseous CO_2 is recovered and, after processing, sent for use in the next cycle, and the oleoresin is precipitated out and sent to the storage device to await use. The described edible gum that serves as the wall material is two or more of acacia gum, maltodextrin, xanthan gum and soluble starch mixed according to a fixed proportion to form an edible gum. The described homogenized and emulsified emulsion is sprayed with a spray gun into a low-temperature atomization dryer for dehydration and drying, the spray gun's incoming spray pressure is 0.2-0.3 MPa and the spray nozzle aperture is 0.8-1.2 mm.

Because the present invention uses spice oleoresin as the core material and it uses room temperature or low-temperature extraction, damage to volatile ingredients in heat-sensitive spices is extremely low and the oxidation reaction can also be controlled to a minimum; when supercritical CO_2 extraction is used in particular it is even less able to produce a chemical reaction in the various components and the extracted oleoresin ingredients are close to the raw materials; using low-temperature atomization drying technology, it is further possible to avoid the partial ingredient damage and oral sensation deterioration that result from high-temperature drying, the clumping caused by vacuum drying, and other drawbacks; in addition to this, the use of a mixture of acacia gum, maltodextrin and other edible gums as the wall material is further able to reduce costs to a certain extent. Thus, the present invention has a reliable

manufacturing technology, a wide range of suitable applications, product ingredients similar to the natural substances, a soft and mellow oral sensation, complementary fragrance and spice flavors, high storage tolerance, high resource use rates, low energy consumption, and other advantages.

Application Example 1

The present application example used the manufacture of ginger oil oleoresin microcapsules as an example. 1. Raw material preprocessing: Fresh mature ginger was used (water content 87%) as the raw material, foreign and poor quality materials were removed, after cleaning and drip drying the weight was 4000 g. Then it was sent into a DS-1 high-speed tissue pulping machine, pulped at 10,000 rpm at 3-min intervals, filtered through a 20-mesh screen to extract the crude fiber and obtain ginger paste; 2. Oleoresin extraction: Using 10 kg concentrated 90% ethanol as the solvent, the ginger paste was immersed 24 h then sent to a continuous percolation device at room temperature using a 5 mL/min flow rate; then the percolation fluid was placed in an HHS constant temperature water bath, vacuum distillation was performed at 8.4-8.5 kPa and 40-45°C to recover the ethanol, to obtain 2520 g hydrous oleoresin (water content 88.44%); after percolation and extraction of the residue the above-described /4 vacuum distillation device was used to recover the ethanol and then it was removed (residue quantity 1980 g); it was combined with the recovered solvent, and reused after 1% activated carbon was used to deodorize it; 3. Emulsification and encapsulation: For wall material the current application example used 29.1 g acacia gum and 261.9 g maltodextrin prepared in combination, 99 g water were added to 2520 g of the above-described hydrous oleoresin in a DS-1 high-speed tissue pulping machine, also at 10,000 rpm, emulsified to form a crude emulsion, at this point in the proportions of crude emulsion:anhydrous oleoresin:edible gum:water = 1:1:8; again the raw material was sent to the JHG homogenizer for homogenization and emulsification at room temperature, the present application

example employed homogenization and emulsification two times, the first time it was homogenized at 10-20 MPa for 6 min and the second time it was homogenized at 30-40 MPa for 9 min to obtain 2910 g of a uniform, stable O/W emulsion (emulsion water content 80%). 4. Dehydration and drying: The above-described emulsion was heated to 45-50°C, pumped into a PGL-3 atomization dryer to perform low-temperature atomization drying; the pump pressure was 0.2 MPa, the spray nozzle aperture 1 mm, the dryer air inlet temperature 80°C, the air outlet temperature 50°, and 558 g powdered fresh ginger oleoresin microcapsules were obtained.

The microcapsules obtained in this application example contained: 3.81% water; the core material retention rate was 96.76%; the yield was 95.88%; the effective ingredient quantity was 1.62%; the ethanol residue was 0.45%; under a Hitachi S-450 electron scanning microscope the mean grain diameter was 39.5 μm.

Application Example 2.

The present application example was used to manufacture garlic oil oleoresin microcapsules as an example:

1. Raw material preprocessing: Fresh garlic was selected (water content 70%), the cloves were separated and the stems were removed. After cleaning and drip drying it too was placed in a high-speed tissue machine and pulped at 10,000 rpm at 3-min intervals, filtered and the peels removed, to obtain 5000 g of garlic paste;

2. Preparation of the oleoresin: 12 kg of the above-described garlic paste was immersed in 55% ethanol as the solvent for 24 h then sent to a continuous percolation device and percolation was performed according to the method in Application Example 1, percolation and recovery of solvent was performed to obtain 3666 g of hydrous oleoresin (water content 85.57%);

3. Emulsification and encapsulation: The wall material used in the current application example was 52.9 g acacia gum and 476.1 g maltodextrin, 37 g of water were added under the same conditions as Application Example 1 to 3666 g of the hydrous oleoresin obtained and emulsified to form a crude emulsion, at this time the proportions of anhydrous oleoresin:edible gum:water = 1:1:6; acacia gum:maltodextrin = 1:9; again a double homogenization method identical to that in Application Example 1 was used to obtain 4232 g of an O/W emulsion (water content 75%);

4. Dehydration and drying: Identical to that in Application Example 1, ultimately 1020 g of powdered garlic oleoresin capsules were obtained. /5

The garlic microcapsules obtained in this application example contained: 7.12% water; the retention rate was 97%; the yield was 96.4%; the effective ingredient quantity was 0.48%; the ethanol residue was 0.42%; the microcapsule mean grain size was 47.25 μm.

Application Example 3.

The manufacture of Sichuan pepper oleoresin microcapsules was used as an example:

1. Raw material preprocessing: Sichuan peppers (water content 12.5%) were selected, cleaned and foreign materials were removed, then they were placed in a DS-1 high-speed tissue pulping machine to pulverize them, and filtered through a 40-mesh screen to obtain 5000 g of Sichuan powder;

2. Oleoresin extraction: 12 kg of the above-described Sichuan peppers were immersed in 55% ethanol as the solvent for 24 h, then sent to a continuous percolation device and percolation was performed at a 5 mL/min flow rate, the percolation fluid and the residue underwent vacuum distillation at 85-90°C and 8.4-8.5 kPa in a HHS constant temperature water bath and the solvent was recovered to obtain 3506 g hydrous oleoresin (water content 57.9%), 6907 g residue (57.9% water content);

3. Emulsification and encapsulation: At proportions of anhydrous oleoresin:edible gum:water = 1:1:5, it was emulsified into a crude emulsion in which the proportions of acacia gum:maltodextrin = 1:9; i.e., 147.3 g acacia gum and 1325.7 g maltodextrin; then, after the crude emulsion was heated to 70°C, a JHG homogenizer was used to perform homogenization twice, and 10311 g of an O/W emulsion (water content 71.42%) were obtained;

4. Dehydration and drying: The dryer in this application example had an air inlet temperature of 90-95°C and an air outlet temperature of 50°C, the rest of the operations were identical to those in Application Example 1, and 2828 g powdered Sichuan pepper oleoresin microcapsules were obtained.

For said microcapsules: 2.98% water, the retention rate was 94.2%, the yield was 96%, the effective ingredient quantity was 9.8%, the ethanol residue was 0.45%, the mean grain diameter was 12 μm.

Application Example 4.

The manufacture of paprika oleoresin microcapsules was used as an example:

1. Raw material preprocessing: After selection, cleaning and pulverization, dry paprika (water content 8.9%) was filtered through a 60-mesh screen to await use;

2. Oleoresin extraction: 5000 g of the above-described paprika powder were sent to an extraction chamber, then, after going through the compressor at 35°C and 30 MPa, 20 kg supercritical CO_2 were sent into the extraction chamber and the extraction was performed, and after 100 min of extraction of the carrier material (paprika powder), the liquid CO_2 was sent into a high-pressure separation chamber to separate the liquid and the residue, after separation of the oleoresin-carrying fluid CO_2, was then placed in a vacuum separation chamber, at 0.1-0.3 MPa and liquid and gas separation was performed; the residue was then discharged by a one-way high-pressure valve into the residue storage tank; after CO_2 /6

was converted to gas in the vacuum separation chamber it was sent to an activated carbon filtration device, the material carried by the adsorbed gas molecules was recovered and sent on to be used in the next cycle, the precipitated oleoresin was sent to the storage device, and 1785 g of hydrous oleoresin were obtained (water content 8.9%);

3. Emulsification and encapsulation: The present application example used 406.5 g xanthan gum and 1219.5 g soluble starch as the wall material, 6345 g of water were added and mixed with 1785 g hydrous oleoresin to form a crude emulsion, then sent to a JHG homogenizer, homogenized at 20-40 MPa at room temperature for 12-15 min, and 9754 g of an O/W emulsion (water content 66.68%) were obtained;

4. Dehydration and drying: 4877 g of the emulsion obtained as described above was used to perform the drying process; the dryer in this application example had an air inlet temperature of 90-95°C and an air outlet temperature controlled to around 50°C, the rest was identical to Application Example 1, 1561 g of powdered paprika oleoresin microcapsules were obtained;

5. Pelleting: 1561 g of the powdered oleoresin obtained served as the medium in a PLG-3 atomization dryer fluidized bed granulation chamber, suspension was promoted to form the fluidized state, then the other half of the 4877 g emulsion obtained was spray-atomized into the granulation chamber to perform pelleting of the powdered microcapsules and drying, to finally obtain 3100 g of grain-form paprika oleoresin microcapsules.

The above-described microcapsules: 4% water, 92.6% retention rate, 95.3% yield, 0.38% effective ingredient, 0.4% ethanol residue, microcapsule mean grain diameter 107.2 μm.